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# **Evaluation of Methods For the Assay of Radium-228 in Water**

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## EVALUATION OF METHODS FOR THE ASSAY OF RADIUM-228 IN WATER

#### by

# J. R. Noyce

The technical literature from 1967 to May 1980 was searched for methods for assaying radium-228 in water. These methods were evaluated for their suitability as potential EPA reference methods for drinking water assays. We suggest the present EPA reference method (Krieger, 1976) be retained but improved, and a second method (McCurdy and Mellor, 1979), which employs  $\beta$  -  $\gamma$  coincidence counting, be added. Included in this report is a table that lists the principal features of 17 methods for radium-228 assays.

Key words: Analysis; assay; evaluation; radioactivity; radiochemistry; radium-228; reference method; water.

#### Introduction

Radium-228 is a radionuclide hazardous to human health because, like radium-226, it is incorporated into the skeleton and has a long biological residence time. Radium-228 is ingested primarily by the drinking of water. It has been found to occur in higher concentrations than <sup>226</sup>Ra in water in contact with some geologic formations in the United States (Johnson, 1971; Michel and Moore, 1980) and in Brazil (Hainberger, 1974). Radium-228 is difficult to assay accurately, especially at low concentrations, because it emits only very low-energy conversion electrons (less than 6 keV) and low-energy beta particles (10 keV average energy). Other isotopes of radium or their daughters cannot readily be added to samples as tracers for measuring the chemical yields of assays because: 1) they are also likely to be present in the water, particularly <sup>224</sup>Ra and <sup>226</sup>Ra, or 2) they are themselves inconvenient to prepare and assay, as in the cases of <sup>223</sup>Ra and <sup>225</sup>Ra. The U. S. Environmental Protection Agency has regulations which set limits on the concentrations of radionuclides, including <sup>228</sup>Ra, in drinking water (EPA, 1976), and which specify approved assay methods. The EPA reference method for <sup>228</sup>Ra (Krieger, 1976) is based on a U.S. Geological Survey method (Johnson, 1971), which was also recommended by the World Health Organization (Lund, 1976). In addition, the U.S. Food and Drug Administration (FDA, 1979) has regulations pertaining to radionuclides in bottled water, modelled on the EPA regulations. Other methods for the low-level assay of <sup>228</sup>Ra are available, but each of them, as well as the present reference method, has one or more deficiencies for use in monitoring.

All assay methods for radionuclides should have sufficient and demonstrated precision, accuracy, and sensitivity to meet the purposes of the assay. With the possible exception of simple screening tests, such as "gross  $\alpha$ " measurements, all radiochemical methods also should incorporate steps for determining the chemical yield.

There are additional desirable features that should be present in an ideal monitoring method. The method should be "rugged", which means it will perform well under less than ideal conditions. It should provide results without requiring a long wait for the ingrowth or decay of a radionuclide, and it should be suitable for use with large numbers of samples; that is, it should have relatively low labor and supply costs.

The Quality Assurance Division in the Environmental Monitoring Systems Laboratory of the EPA, Las Vegas, requested the NBS Radioactivity Group, Nuclear Radiation Division, to survey the literature for <sup>228</sup>Ra assay methods and evaluate their suitability as potential EPA reference methods.

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### Survey of Literature

Nuclear Science Abstracts was searched from 1967 until its termination in 1977. Then, the Energy Data Base was searched from 1977 through May 1980. Also, Chemical Abstracts was searched from 1968 through May 1980. References cited in the relevant papers found in these searches led to other papers dating from approximately 1960.

There are only a few papers giving methods for assaying <sup>228</sup>Ra in water and the results of such measurements. Many of the available papers deal with seawater rather than fresh water. The pertinent aspects of the methods are set forth in Table 1 in alphabetical order of author. Some of these publications also deal with <sup>226</sup>Ra assays, and salient features of these methods are included in Table 1. Papers concerned only with <sup>226</sup>Ra assays are not included.

Also in Table 1 are <sup>228</sup>Ra assay procedures found during the search that are for other types of samples, such as soil. The latter methods usually can be adapted to the assay of water samples, and some contain useful techniques not exploited in the water-assay procedures. Reports of investigations using previously published <sup>228</sup>Ra assay procedures are excluded.

# Evaluation of Methods

Most of the following discussion further explains important points made in Table 1. Nearly all the assay methods separate and purify <sup>228</sup>Ac and count beta particles emitted in its decay. The method of McCurdy and Mellor (1979) is unique in that the <sup>228</sup>Ac is not separated from its <sup>228</sup>Ra parent for counting. Thus, <sup>228</sup>Ac can be counted as long as desired or recounted days later without performing additional radiochemistry on the sample. A few procedures use the detection of radiations emitted by later members in the  $^{228}$ Ac decay series, including  $^{228}$ Th,  $^{224}$ Ra, and even  $^{212}$ Pb from de-emanated  $^{220}$ Rn. Only the method of Tomza (1977) uses liquid-scintillation counting.

The assay methods listed in Table 1 appear to have the sensitivity required by the EPA drinking water regulations (EPA, 1976), with the apparent exceptions of those of Iyer <u>et al</u>. (1966) and Baretta and Feldman (1961). There is some evidence that at low  $^{228}$ Ra concentrations a method for counting alpha particles from the decay of  $^{228}$ Th has better precision than one for counting beta particles from the decay of  $^{228}$ Ac (Knauss <u>et al</u>., 1978), but the time required for  $^{228}$ Th ingrowth is much longer than that for  $^{228}$ Ac.

The methods in Table 1 lacking a provision for determining the chemical yield for each sample processed are those of Humphrey <u>et al</u>. (1975), Johns <u>et al</u>. (1979), Kuchta <u>et al</u>. (1976), MacKenzie <u>et al</u>. (1979), Percival and Martin (1974), Petrow <u>et al</u>. (1964), Sakanoue <u>et al</u>. (1973), and Tomza (1977). This is also true of procedures of Barratta and Feldman (1961), and of Kahlos and Asikainen (1973), but both could readily be modified for gravimetric determination of chemical yields. Some of the methods which do provide a chemical yield, however, require long waiting times (see below).

Four of the methods use radioactive tracers to measure chemical yields: Kaufman <u>et al</u>. (1973), Koide and Bruland (1975), Michel and Moore (1980), and Smith and Mercer (1970). Because the present EPA reference methods for radionuclides other than tritium do not require radioactive tracers (Krieger, 1976), it was assumed they are to be avoided when another yield-measuring technique

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is available. It should be noted that the chemical yield is incorrectly calculated in the reference method for  $^{228}$ Ra (Krieger, 1976). Any loss of sample in steps 13 and 14 is counted twice. The Ba(Ra)SO<sub>4</sub> precipitate at the end of step 11 should be weighed, rather than in step 25 as stated.

The waiting periods in the methods given in Table 1 for ingrowth or decay of radionuclides during an assay can be divided into three groups: less than two full days, two days to 30 days, and greater than 30 days. Waiting periods include time for the ingrowth of  $^{222}$ Rn for those procedures where assays of  $^{226}$ Ra are an integral part of the  $^{228}$ Ra assays. The shorttime procedures in the first group are Baratta and Feldman (1961), Humphrey <u>et al</u>. (1975), Iyer <u>et al</u>. (1966), Krieger (1976), McCurdy and Mellor (1979), Percival and Martin (1974), and Petrow <u>et al</u>. (1964). Methods with waiting periods of between two and 30 days are Johns <u>et al</u>. (1979), Kahlos and Asikainen (1973), Koide and Bruland (1975), and Smith and Mercer (1970) (<u>via</u>  $^{228}$ Ac). Methods requiring waiting times of longer than 30 days are Kaufman <u>et al</u>. (1973), Kuchta <u>et al</u>. (1976), MacKenzie <u>et al</u>. (1979), Michel and Moore (1980), Sakanoue <u>et al</u>. (1973), Smith and Mercer (1970) (via  $^{228}$ Th), and Tomza (1977).

The methods of Kuchta <u>et al</u>. (1976) and Tomza (1977) would have waiting times of only two to 30 days if  $^{224}$ Ra were known to be in equilibrium with  $^{228}$ Ra, but this condition would probably be rare for drinking water. Finally, the waiting period in the procedure of MacKenzie <u>et al</u>. (1979) could also be reduced to between two and 30 days if the more volatile beta-particle-emitting progeny of  $^{228}$ Ac could be flamed off a

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source evaporated on a stainless steel planchet with no accompanying loss of <sup>228</sup>Ac (compare with Koide and Bruland (1975)).

A potential problem confronting the methods which assay  $^{228}$ Ra  $\underline{via}$   $^{228}$ Ac is the presence of radioisotopes of the rare-earth elements or those with similar chemical properties, especially  $^{90}$ Y, daughter of  $^{90}$ Sr. Some of the procedures have one or more steps designed to reduce such contamination, and the  $\beta$ - $\gamma$  coincidence counting technique of McCurdy and Mellor (1979) is not affected by  $^{90}$ Y. A recent interlaboratory assay exercise, with a water test sample that had  $^{90}$ Sr- $^{90}$ Y added, indicated that the present EPA reference method does not adequately remove  $^{90}$ Y from  $^{228}$ Ac (Whittaker, 1980).

The Y or La compound chosen as the carrier for Ac should be checked for radiochemical purity before use, because it may be a significant source of beta activity (Johnson, 1971). Such a warning should be added to the reference method.

It is difficult to assess the "ruggedness" of an analytical method from just a literature survey. In general, the less complex an assay procedure is, the better chance of its being "rugged". However, some of the simpler procedures in Table 1 are without a means for measuring chemical yields.

The "special apparatus" columns in the table list items needed for each method that are judged not likely to be present in the usual water-analysis laboratory. Some of these items, such as the  $\beta - \gamma$  coincidence-counting system of McCurdy and Mellor (1979), are a significant expense.

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## Conclusions

Most of the  $^{228}$ Ra assay methods in Table 1 are eliminated from consideration as an EPA reference method by applying the criteria discussed earlier: adequate sensitivity, yield determination without radioactive tracers, and a waiting time for ingrowth or decay of radionuclides in the sample of no more than 30 days. The methods meeting these criteria, but with the reservation already noted about each, are Kahlos and Asikainen (1973), Krieger (1976) and McCurdy and Mellor (1979). The first of these three would be eliminated if the requirement of a waiting period for ingrowth or decay of no more than two days were imposed. It is possible that Iyer <u>et al</u>. (1966) and Barratta and Feldman (1961) are sufficiently sensitive, but multilaboratory studies would be necessary to demonstrate this.

It is suggested that the EPA keep Krieger (1976) as a reference method but improve its decontamination factor for  $90\gamma$  and correct its chemical yield calculation. It is also suggested that McCurdy and Mellor (1979) be added as a second reference method. The two procedures complement each other. The first uses radiation-detection systems which most laboratories that assay for radionuclides in water are likely to have, but it requires a considerable amount of labor to process samples. The second requires most laboratories to purchase a new radiation-detection system, but the labor to process samples is definitely less.

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McCurdy, D.E., and R.A. Mellor (1979), "An alternative technique for the determination of  $^{226}$ Ra and  $^{228}$ Ra in drinking water", presented at 24th ann. meeting of Health Physics Soc., July 8-13, 1979, Philadelphia.

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Sekine, T., Y. Koike, and M. Sakairi (1966), "Studies of actinium (III) in various solutions, (I) Separation and purification of <sup>228</sup>Ac in <sup>228</sup>Ra solutions by solvent extraction method", J. Nucl. Sci. Tech., 4, 308.

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The following papers were obtained too late to include in Table 1. They do not alter the conclusions of this report because none is suitable as a reference method for radium-228 assays. The radiochemical section of the Herment paper deals with the production of radium-228. Neither of the other two papers has a way to determine the chemical yield of an assay.

Herment, M. (1971), <u>Chemical Separation and High Resolution Nuclear Spectros-</u> <u>copy in the <sup>2 2 8</sup> Ra <sup>2 2 8</sup> Ac <sup>2 2 8</sup> Th Radioactive Chain</u>, Univ. Grenoble Report FRNC-TH-184 (in French).

Kharbanda, J.L., M.V. Rajan, R.V. Amalraj, K.T. Thomas (1966), "Separation and estimation of radium-228 in thorium plant effluent", in <u>Proc. Nucl. Radiat.</u> <u>Chem. Symp.</u>, Jan. 20-22, 1966, Waltair, p. 46.

Sebessy, L., E. Upor, and I. Vados (1967), "Determination of the radium-226/ radium-228 ratio in natural waters", in Proc. Conf. Appl. Phys.-Chem. Methods Chem. Anal., 1966, Budapest, Vol. 2, p. 245. Table 1. Comparison of Major Features of Methods for the Assay of Radium-228 in Water

						R A	0 I U M - 2 2 6 -	
Reference	Intended Use	Sample Size	Preliminarjes	Ra Separation	Purification	Final Form	Yielded By	Counted 8y
Baratta and Feldman (1961)	Assay of Ac in U mill effluents and natural wa- ters	l liter	Ac pptd. with La (12 mg) by HF					
Humphrey <u>et</u> <u>al</u> . (1975)	<sup>226</sup> Ra and <sup>228</sup> Ra in effluent so- lutions with high ion concentration		None	On Ba(NO <sub>3</sub> )2 in 70% HNO <sub>3</sub> . 2Sul- fate ion inter- feres	By AG50W-X8 ion exchange column operated at 60°. Eluted with 3 M HNO <sub>3</sub>	Solution evap. on stainless steel planchet, planchet flamed	Nothing	Frisch-grid ioni- zation chamber of 4.78 MeV < with PHA
lyer <u>et</u> <u>al</u> . (1966)	Estimation of Ra in mona- zite and Th sam- ples	2 to 5 g	Oigest with H <sub>2</sub> SO <sub>4</sub> in Pt dish and fuse with Na <sub>2</sub> CO <sub>3</sub> and K <sub>2</sub> CO <sub>3</sub> . Oissolve in FNO <sub>3</sub>	On 100 mg of Ba pptd. as BaCO <sub>3</sub> . 8a weighed be- fore and after Ra separation				
Johns <u>et</u> <u>al</u> . (1979)	226 <sub>Ra</sub> and 228 <sub>Ra</sub> in water, soil, air, and biolog- ical samples	1500 g water, l g soil,2up to 500 cm of air filter, biological not specified	Water, none; di- gest soil in Paar bomb, then fuse; acid dis- solution of air filters	Water, air fil- ters: coppt. on PbSO <sub>4</sub> ; soil: co- ppt. on 8aSO <sub>4</sub>	Water and Micro- sorban filters: none; soil and glass-fiber fil- ters: reppt. on BaSO <sub>4</sub>	Water and Micro- sorban filters: OPTA complex in acetic acid; others: chloride solution	Nothing	Rn de-emanated into scintillation cham- ber after 30 days ingrowth. Count- ing begun 4.5 h af- ter transfer
Kahlos and Asikainen (1973)	Assay of natural radioactivity in ground water	l liter	Citric acid and NH <sub>4</sub> OH added to filtered sample, then Ba and Pb carriers	Mixed sulfates pptd. with H <sub>2</sub> SO <sub>4</sub>	Oissolve ppt. in alkaline EOTA solution, reppt. sulfates at pH 4.5 with acetic acid	8a(Ra)SO <sub>4</sub> on planchet	Weighing of 8a(Ra)SO <sub>4</sub> ppt.	ZnS(Ag)-activated ≪-partlcle scin- tillation counter
Kaufman <u>et</u> al. (1973), 8roecker et <u>al</u> . (1973)	<sup>228</sup> Ra and <sup>228</sup> Th in sea water	600 to 800 1	Acidify with HCl, add Fe and $230$ carriers, 230 Th; ppt. Fe(OH) <sub>3</sub> and Ba(Ra)SO <sub>4</sub> with NH <sub>4</sub> OH	Hydroxides dis- solved in HCl; sulfates trans- posed to carbon- ates, dissolved in HCl	None	Rn de-emanated by He gas. Rn frozen out of He in liquid air- cooled trap	Nothing. Moore (1969b) found initial sulfate pptn. to be 60% to 90% efficient	≪ -scintillation detector
Koide and Rruland (1975)	226 <sub>Ra</sub> , 228 <sub>Ra</sub> and other natural emitters in sea water and marine sediments	20 1 water, 1 to 5 g sedi- ment	Add Pb carrier, 22 Ra tracer. Sediment: ignite and leach with HCl	Water: coppt. on AIPO <sub>4</sub> , dissolve in 4 <sup>M</sup> HNO <sub>3</sub> . Sediment: coppt. on Pb(NO <sub>2</sub> ) <sub>2</sub> with <b>75% HNO<sub>3</sub></b>	Separate Ra and Pb on anion ex- change column, alkaline earths on cation ex- change column	Electroplated on Pt planchet af- ter at least two weeks wait for upgupported Ra to decay	223Ra tracer. 227Ac "cow" is required	✓-spectrometry with semiconduc- tor detector
Krieger (1975), based on Johnson (1971)	<sup>226</sup> Ra and <sup>228</sup> Ra in drinking wa- ter	At least l liter	Add citric acid, Pb, Ba, and Y carriers; heat	Add NH <sub>4</sub> OH, ppt. mixed Sulfates with $H_2SO_4$ , then add (NR <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	Multiple copptn. on Ba(NO <sub>3</sub> ) <sub>2</sub> and on BaSO <sub>4</sub>	In aklaline EOTA solution, or in 8aSO <sub>4</sub> ppt. on stainless steel planchet	BaSO <sub>g</sub> gravimet- rically	Rn de-emanation into scintillator cell
Kuchta <u>et</u> <u>al</u> . (1976)	Assa¥28f skele- tal <sup>228</sup> Th in pres- ence of gross amounts of <sup>226</sup> Ra	Not speci- fied	Ory-ash bone sample, dissolve ash in HNO <sub>3</sub>	None				

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Special Items	Purification	Final Form	Yielded 8y	Waiting Period	Counted By	Special Items	Comments
	Liquid-liquid extractions us- ing TTA in ben- zene; ion ex- change columns	La(Ac) oxalate. Iyer <u>et al</u> . (1966) think Y is better than La as carrier	Not mentioned, but could be gravimetrically	None stated, but sample should be two days old be- fore starting assay	G-M tube or pro- portional coun- ter, with absorb- er when Ac is present	None	Sekine <u>et al</u> . (1967) say TTA equimolar with TBP in CCl <sub>4</sub> is superior to TTA only
Heated ion ex- chagge columns (60°C)	2nd ion exchange column at 60 <sup>0</sup> C. Ac eluted with 6 M HNO <sub>3</sub>	Solution evap. on stainless steel planchet	Nothing	24 h for <sup>228</sup> Ac ingrowth	Gas-flow propor- tional counter with 13 mg/cm absorber	Same as for ZZO <sub>Ra</sub>	Ba initially present in samples. Na, K, Ca, Mg, and Al each more than 100 mg/l in typical sam- ple
	Ac copptd. on Fe(OH) <sub>3</sub> , impuri- ties scavenged on Pb and 8i sul- fides	Fe(Ac)(OH) <sub>2</sub> dis- solved in HNO <sub>2</sub> . Known fraction evap. on plan- chet	Weighings of ppt. containing Ra an Ac, or spectro- photometric as- say of Fe	24 h for <sup>228</sup> Ac ingrowth	End-window, gas- flow proportion- al counter	Pt crucible; spectrophotome- ter (Fe line at 304 nm used)	Method not tried on very low activity samples
Paar bomb; Pt dish for glass- fiber filter and soil samples	Reppt. 8a(Ra)SO <sub>4</sub> , dissolve, ex- tract Ac into OEHPA, back ex- tract into 1 M HNO <sub>3</sub>	Evap. acid solu- tion to dryness on 5-cm diameter planchet	Nothing	About 30 d for 222 Rn ingrowth; at least 30 h for 228 Ac in- growth	Low-background proportional counter. Time T <sub>3</sub> is ambiguous- ly specified	None besid <u>9</u> 26 <sub>Ra</sub> those for	Method could be changed to assay <sup>228</sup> Ra first. No specifics given for as- saying biologicals. Sec- tion 11 wrongly states no "Ra std. are available
None. Method based on that of Goldin (1961)	Oissolve ppt., add Ce carrier, transfer to depo- sition cell, add Te; Pb, Bi, and Po plate out.	Ppt. Ce with NaOH, dissolve ppt. in HNO <sub>3</sub> , add 8a holdBack carrier, reppt. Ac on Ce(OH) <sub>3</sub>	Nothing, but final ppt. could be Ce oxalate, which is weigh- able for yield	Abgut 30 d for Rn ingrowth	Low-background Ø counter, type unspecified	Constant temper- ature bath, for the spontaneous electrodeposi- tion step	<sup>226</sup> Ra could be counted by de-emanation of <sup>222</sup> Rn
Liquid air- cooled trap for Rn	Th copptd 238n $Fe(OH)_{3}$ ; Th spike added, so- lution stored; Th copptd. on $Fe(OH)_{3}$	Th purified on cation and anion exchange col- umns, extracted into TTA in ben- zene, evap.	226 <sub>Ra</sub> assays of sample initially and during <sup>228</sup> Th ingrowth plus - £96ctrometry of Th tracer	4 to <sub>2</sub> 11 months for 228 Th in- growth	✓ spectrometry; counting system not specified	None additional	Method is based in part on Moore (1969a). Knauss et al. (1978) found pre- cision of assays better by countig28 Ac
<sup>227</sup> Ac "cow". Ul- trasonic agita- tor	See under <sup>226</sup> Ra; none additional	On same planchet as ZZBRa	223 <sub>Ra</sub>	At least 2 weeks for 224 Ra and progeny to decay	228 <sub>Ac in propor- tional counter with Al absorber. Other emitters must be flamed off planchet</sub>	None additional	$\begin{array}{c} 228_{Ra}/226_{Ra} \text{ activity ra-}\\ \text{tio must be at least 0.1.}\\ \text{As check, ingrown $T$}\\ \text{can be counted later.}\\ \text{Cochran (1979) conj-letely}\\ \text{dissolved sediments.} \end{array}$
None	As for <sup>226</sup> Ra plus copptn. of ingrown Ac on Y(OH) <sub>3</sub>	In Y oxalate ppt.	Weighing of Y oxalate	At least 36 h ž28 ingrowth of Ac	228 <sub>Ac</sub> in gas- flow proportion- al counter	None	Chemical yield of Ac is incorrectly calculated — see text
	None except by Rn de-emanation using a flow- through collec- tion chamber	Solution is26e- emanated, 226en and progeny col- lected on nega- tively charged electrode	Nothing	2 counts about 1 year apart for samples without equilibrium be- twgen 228 Ra and 228 Th	Electrode placed in holder with ZnS-coated Mylar disc op photo- tube. Pb and progeny counted	Flow-through chamber with collection elec- trode; holder for electrode on phototube	Data are analyzed by com- puter. Method designed to have poor sensitivity for ZCRa

				г		R A	DIUM-226-	
Reference	Intended Use	Sample Size	Preliminaries	Ra Separation	Purification	Final Form	Yielded 8y	Counted By
MacKenzie <u>et</u> <u>al</u> . (1979)	Assay of <sup>228</sup> Ra and other radio- nuclides in sea water and marine sediments	Water: 1D to 226 <sub>Ra28</sub> 1DDD 1 for Ra. Sediment: S to 1D g	Water: filter, acidify. Sedi- ment: complete dissolution in Teflon with mix- ture of acids	226Ra: none. 228Ra: absorb on MnD <sub>2</sub> , desorb with HCl, coppt. on BaSD <sub>4</sub>	None	222 <sub>Rn gas</sub>	Nothing, but au- thors say assay is quantitative	∝ -scintillation in modified Lucas cell
McCurdy and Mellor (1979)	<sup>226</sup> Ra and <sup>228</sup> Ra in drinking wa- ter	2 1	Add citric acid, adjust pH to 9.S, add 8a carrier (known amount)	2 4	Dissolve ppt. in alkaline EDTA solution, reppt. BaSD <sub>4</sub> with ace- tic acid	BaSD, ppt. mixed with ZnS powder. ZnS/BaSD, ratio is approximately 2.4	Weighing of BaSO <sub>4</sub> ppt.	≪-¥coincidense counting of <sup>226</sup> Ra
Michel and Moore (198D)	Assays of <sup>226</sup> Ra and <sup>228</sup> Ra in ground waters	226 <sub>Ra:</sub> D.8 1; 228 <sub>Ra:</sub> 1D to 2D 1	None (waters assayed were clear)	226 228 Ra: absorb on MnO <sub>2</sub> -impregnated acrylic fibers, desorb with HCl, ppt. on 8aSD <sub>4</sub>	None	<sup>222</sup> Rn gas	Nothing, but each sample run at least twice	≪-scintillation chamber
Percival and Martin (1974)	<sup>226</sup> Ra and <sup>228</sup> Ra in soils, water, ores, and mill tailings and effluents	S g of solids, D.l to l li- ter of liquid	Liquids: acidify, wait overnight, coppt. on PbSD <sub>4</sub> , do sulfate fu- sion	Add acetic acid, coppt. Ra with BaCl, on BaSD <sub>4</sub> . Dissolve ppt. in DTPA solution	None	<sup>222</sup> Rn gas	Nothing	Pulse-ionization chamber or <- scintillation cell
Petrow <u>et</u> <u>al</u> . (1964)	Assa¥28 <sup>f 228</sup> Ra and ¥28 <sup>Th</sup> in biological and mineral samples	Up to SD g of bone ash; up to 2D g of food or plant ash; S g of minerals	Minerals: fuse with Na <sub>2</sub> CD <sub>3</sub> , di- gest in NaOH. All types: dis- solve in acid	Liquid-liquid extractions with Aliquat 336 and DEHPA to remove Th. Coppt. Ra on PbSD <sub>4</sub>				
Sakanoue <u>et</u> <u>al</u> . (1973)	228 <sub>Ra</sub> in s <b>e</b> a water and <sup>231</sup> Pa in sea sedi- ments	80 1	BaCl <sub>2</sub> and FeCl <sub>3</sub> added, then $\mathrm{NH}_3$ . Fe(OH) <sub>3</sub> and BaSO <sub>4</sub> ppt.	Fe(DH) <sub>3</sub> ppt. dis- solved <sup>3</sup> with HCl. 8a(Ra)SD <sub>4</sub> con- verted to car- bonates by fu- sion, dissolved in HCl				222 <sub>Rn</sub> de-emanation (no specifics giv- en)
Smith and Mercer (197D)	Assay28f <sup>226</sup> Ra and <sup>228</sup> Ra in soils and plants	Up to 1D g of plant ash and soil	Repeated evap. with HClD <sub>d</sub> and HF, dissolve residue in HCl	Coppt. Ra on Ba and Pb sulfates. Ppt. dissolved in alkaline EDTA sulfates repptd. with acetic acid	Cation exchange separation of Ra and Ba; Ra coppt. on Pb(ND <sub>3</sub> ) <sub>2</sub> , sep arated by anion exchange at 60	Electroplated on stainless steel planchet	133Ba for disso- lution only, 225Ra for entire 258ay, from Th "cow"	← -spectrometry with Frisch-grid pulse- ionization chamber
Tomza (1977)	Assay of low Ra concentrations in mineralized waters	10 1	Add citric acid and NH <sub>4</sub> DH, then Pb and 8a carri- ers	Mixed sulfates pptd. with H <sub>2</sub> SD <sub>4</sub>	Ppt. dissolved in alkaline EDTA then repptd. by acetic acid at pH 4.5	8a(Ra)SD <sub>4</sub> ppt. dispersed in toluene contain- ing PPD, PDPDP, and colloidal silica	Nothing	Liquid-scintilla- tion, two counts several weeks apart

ABBREVIATIONS:  $\alpha = \alpha$  particle  $\beta = \beta$  particle coppt. = coprecipitate DEHPA = bis(2-ethylhexyl)phosphoric acid

DTPA = sodium diethylenetriaminepenta acetate EDTA = sodium ethylenediaminetetraacetate evap. = evaporate, evaporated G-M = Geiger-Müller

PHA = pulse height analyzer PDPDP = 1,4-di-(2-(S-phenyloxazoyl))-benzene PP0 = 2,5-diphenyloxazole ppt. = precipitate (as verb and as noun)

	•		RADIU	M - 2 2 8			
Special Items	Purification	Final Form	Yielded By	Waiting Period	Counted 8y	Special Items	Comments
25-liter glass vessel for equi ibration of Rn in water sample liquid N <sub>2</sub> -cooled trap for <sup>2</sup> Rn	Ac by TTA in ben- zene; store 5	Evap. organic phases on heated stainless steel planchet	After <sup>228</sup> Ac as- says, sample al- 29cassayed for CRa. Extrac- tion of Ac is not yielded	12 d for <sup>222</sup> Rn ingrowth, at least 5 weeks fag decay of Th progeny	Tracerlab Omni- guard (gas-flow G-M counter with anticoincidence)	MnO <sub>2</sub> -impregnated acrylic fibers, Pt crucible	Method dogsgnot discuss assay of Ra in sedi- ments
<pre>4 timing SCA, 2 coincidence ana lyzers, special detector as- sembly, PHA</pre>	None additional. See under Ra	Same as <sup>226</sup> Ra	Same as <sup>226</sup> Ra	<sup>228</sup> Ac ingrowth time not speci- fied, assumed to be 1 to 2 days	β-δ coincidences Segnting of 228 <sub>Ac</sub>	Same as for 220 Ra	Considerable instrumen- tation is required, but amount of radiochemis- try is reduced
Apparently none	Carbonate fusion dissolve mixed ppt. in HCl, co- ppt. Th on Fe store, add coppt. Th on Fe	exchange, mount on planchet for	A spectrometry of Th, Ra as- say of concentra- ted sample. Sep- arate 20 Ra as- say also needed	2220 10 d for 222Rn ingrowth, 2 to228months for Th in- growth	<pre>     Spectrometry     950     Th and     Th; counting     system not spec-     ified </pre>	MnO <sub>2</sub> -impregnated acr9lic fibers. See Moore (1976) for preparation	Ra extraction by fibers based on Krishnaswami <u>et</u> al. (1972) and on Moore and Reid (1973)
Pt dish	Remove 8a as sul- fate, extract Ac from supernate into 15% DEHPA, strip into HNO <sub>3</sub>	- Add Ce, coppt. Ac on Ce exalate	Nothing	At least 20 h for <sup>228</sup> Ac in- growth	Gas-flow propor- tional counter	None additional	
	Metathesize ppt. to carbonates, dissolve, coppt. Ra on $Pb(NO_3)_{2}$ ; extract Ac with DEHPA, Aliquat	Evap. final acid solution on 5- cm diameter stainless steel planchet	Nothing	At least 36 h for <sup>220</sup> Ac in- growth	228 rom decay of Ac. Instru- ment unspecified	Pt crucible	This method is extention of similar method by Petrow and Allen (1961) for U mill effluents which also is unyielded
	Extract Ac into DEHPA, back ex- tract into HNO <sub>2</sub> , load on Doxex 50 ion exchange col- umn, elute with 6 M HNO <sub>3</sub>	Evap. on stain- less steel plate	226 <sub>Ra</sub> assay on initial sample and on Ba(Ra)Cl <sub>2</sub> solution just be- fore Ac extrac- tion. Extraction is <u>not</u> yielded	Unspecified. As- sume 27 d 59% each of 2 <sup>292</sup> Rn ingrowths	228ounting of Ac in guarded, gas-flow propor- tional counter, or in fijitsu "pico beta" β spectrometer	None	
Heated ion ex- shange column; 133Th "cow"; 133Ba tracer	Coppt. Ac on Fe, solution stored, Ac reppt. on Fe, Ac separated on anion exchange column from Fe	Solution evap. on stainless steel planchet	Espounting of Ac and proge- ny. 13 Ba not needed if method used with water sample	7 to 10 d £95 growth of <sup>225</sup> Ac	Ac and Ac in gas-flow pro- portional coun- ter with 13 mg/ cm absorber	None additional	Alternate <sup>228</sup> Ra method:
None	Simul tageous with tage Ra	Same as for	Nothing	Equilibrium be- tygen <sup>220</sup> Ra and <sup>21</sup> Th assumed. If not so, must reassay several months later	Liquid-scintilla- tign counting of Ra and proge- ges & pulses of Ra discrimina- ted against	None	Use of moderately com- plex equations of in- growth and decay is re- quired

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reppt. = reprecipitate SCA = single channel analyzer std. = standard TBP = tributylphosphate TTA = 2-theonyltrifluoroacetone

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Document describes a computer program; SF-185, FIPS Software Summary, is attached. 11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here) The technical literature from 1967 to May 1980 was searched for methods for assaying radium-228 in water. These methods were evaluated for their suitability as potential EPA reference methods for drinking water assays. We suggest the present EPA reference method (Krieger, 1976) be retained but improved, and a second method (McCurdy and Mellor, 1979), which employs $\beta$ - $\gamma$ coincidence counting, be added. Included in this report is a table that lists the principal features of 17 methods for radium-228 assays.								
<ol> <li>KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons) Analysis; assay; evaluation; radioactivity; radiochemistry; radium-228; reference method; water.</li> </ol>								
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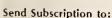
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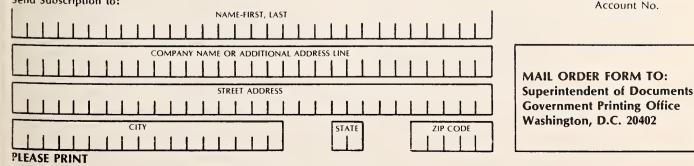
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